

# Formation of Nanoporous Poly(methyl silsesquioxane) Thin Films using Adamantane for Low-k Application

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**ABSTRACT:** Nanoporous poly(methyl silsesquioxane) PMSSQ thin films for low-k application have been prepared using chemically attached adamantylphenols as pore generating materials (porogen). To obtain the mechanically stable porous structure, multifunctional 1,2-bis(trimethoxysilyl)ethane (BTMSE) was employed in addition to methyltrimethoxysilane as a main matrix material. From the decomposition of porogen, confirmed by FTIR and TGA, the nanoporous thin films containing pores less

than 5 nm, which are characterized using sorption analysis, were successfully achieved. The dielectric constant was significantly decreased to 1.9, while maintaining the stable mechanical properties with the elastic modulus of 3.7 GPa measured by a nanoindenter. © 2007 Wiley Periodicals, Inc. *J Appl Polym Sci* 104: 2906–2912, 2007

**Key words:** synthesis; thin films; structure; dielectric properties; templates

## INTRODUCTION

Poly(silsesquioxane) (PSSQ) materials have recently acquired much attention in both fundamental research and applied science. PSSQs, represented by the empirical formula  $(\text{RSiO}_{3/2})_n$  may form a product composed of various structures including cage, ladder, and their polymers during sol-gel polymerization.<sup>1</sup> This diverse structural evolution has attracted much interest in basic research fields. In addition, owing to their exceptional electrical, thermal, and oxidative stability, PSSQs are extensively used in promising applications such as low dielectric (low-k) insulators for microelectronic devices, nanocomposites as a reinforcing element, and nanoreactors for synthesis of nanoparticles.<sup>2–4</sup>

Among various PSSQs, the poly(methyl silsesquioxane) (PMSSQ) for low-k applications has been intensively investigated because of an intrinsic low dielectric constant ( $k = 2.7\text{--}3.0$ ), low moisture absorption, and high thermal stability to 500°C when fully cured. The dielectric constant of PMSSQ thin films could be lowered further by the introduction of air-filled porosity. For instance, nanoporous structures were realized into PMSSQ thin films through the elimination of thermally labile pore generating materials (porogens) imbedded in films. A variety of porogens ranging from nanoscopic molecules to macromolecules, which were blended with or covalently

bonded to matrix materials, were used to reduce the dielectric constant of PMSSQ thin films and a low-k value as low as 1.5 was successfully achieved.<sup>5–12</sup>

Nevertheless, the inherent low mechanical properties of low-k dielectrics due to the reduced film density should further be overcome for successful low-k implementation. For instance, under harsh process such as chemical mechanical planarization (CMP), low-k dielectrics often suffer from the destruction of nanoporosity within the film or delamination.<sup>13</sup> Besides, as shown in our previous results, the introduction of graft-type porogen facilitates the collapse of porous structures with porogen loading, resulting in the increase of dielectric constant and refractive index.<sup>14</sup> In contrast, when bridge-type porogen having the six Si–OH groups fully hydrolyzed was used, the increase in the dielectric constant and refractive index was not observed, showing the desirable low-k values and mechanical properties. These results indicate that the multifunctional silica sources such as 1,2-bis(trimethoxysilyl)ethane (BTMSE) or tetraethoxyorthosilicate (TEOS) can reinforce mechanical strength of dielectric thin film without a significant increase of low-k value.<sup>15,16</sup>

In this study, it is presented that nanoporous PMSSQ thin films with low dielectric constant were successfully prepared through the thermal decomposition of graft-type adamantylphenol porogens covalently attached to the PMSSQ matrix and their mechanical properties were stabilized by the introduction of BTMSE. Organosilicates composed of BTMSE and methyltrimethoxysilane were also used as main

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matrix materials and their thin film properties were investigated with porogen loading.

## EXPERIMENTAL

### Materials

All the reagents used in present study were purchased from Aldrich and TCI and were used as received unless otherwise specified.

### Synthesis

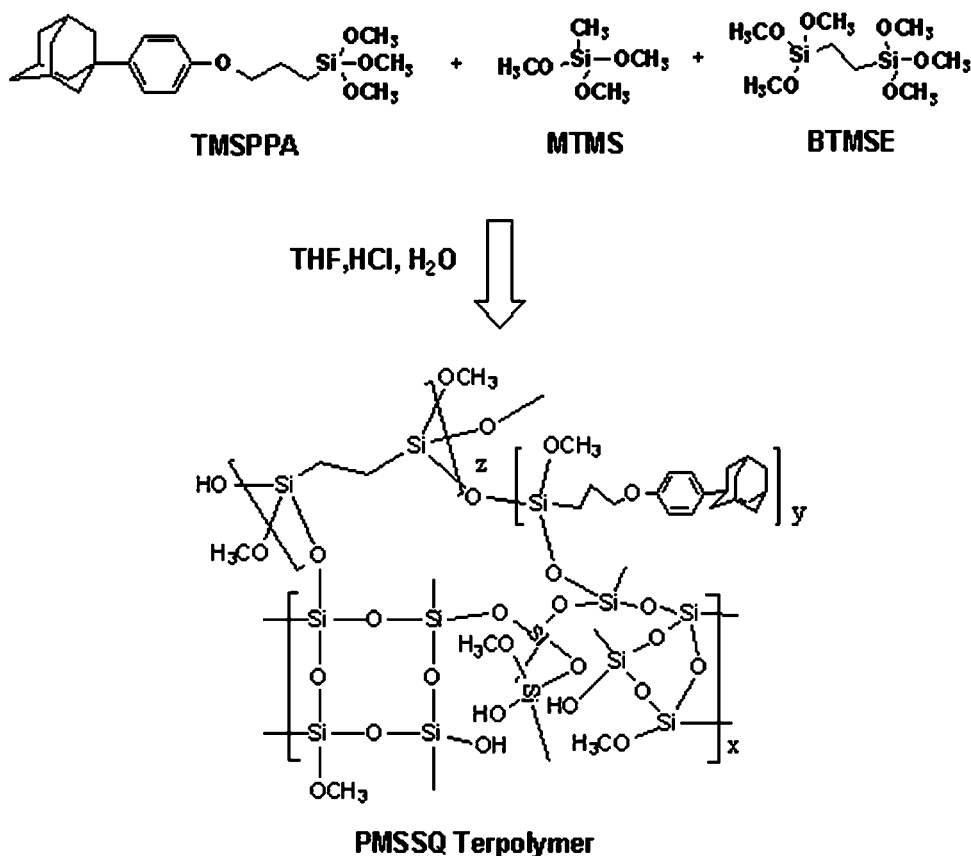
The alkoxy silane precursor having adamantylphenol porogen was synthesized according to the method reported previously.<sup>13</sup> As shown in Figure 1, a typical synthesis procedure of PMSSQ terpolymer with BTMSE of 20 mol % and adamantylphenol porogen is shortly described here; 1-4-(trimethoxysilylpropoxyphenyl)adamantane (TMSPPA) (2.0 g, 2.6 mmol), 1,2-bis(trimethoxysilyl)ethane (BTMSE) (1.4 g, 5.3 mmol), methyltrimethoxysilane (MTMS) (2.5 g, 18.2 mmol), and THF (14.2 g) were added into a 50-mL three-neck, round-bottom flask. At 25°C, HCl dissolved in H<sub>2</sub>O was added dropwise into the vigorously stirred reaction mixture over 30 min. After 30 min, the

reaction mixture was refluxed at 60°C for 10 h, cooled to room temperature, and 30 mL of diethyl ether was added. The organic layer was extracted with H<sub>2</sub>O (3 × 30 mL) and was dried over Na<sub>2</sub>SO<sub>4</sub>. The solvent was evaporated off under reduced pressure to yield a 1.84 g in white solid form. <sup>1</sup>H NMR (*d*-Acetone):  $\delta$ 7.26 (br, -ArH-, 2H), 6.85 (br, -ArH-, 2H), 5.74 (br, -OH, 1H), 3.95 (br, -CH<sub>2</sub>-, 2H), 3.48 (br, -OCH<sub>3</sub>, 3H), 2.06 (adamantyl, -CH-, 3H), 1.89 (adamantyl, -CH<sub>2</sub>-, 6H), 1.78 (adamantyl, -CH<sub>2</sub>-, 6H) 0.85 (br, -CH<sub>2</sub>-, 6H), 0.83 (br, -CH<sub>2</sub>CH<sub>2</sub>-, 4H), 0.13 (br, Si-CH<sub>3</sub>, 3H).

As a reference sample, PMSSQ without porogen was also prepared using the same reaction condition described above. <sup>1</sup>H NMR (*d*-Acetone):  $\delta$ 5.76 (br, -OH-, 1H), 3.50 (br, -OCH<sub>3</sub>-, 3H), 0.85 (br, -CH<sub>2</sub>CH<sub>2</sub>-, 4H), 0.12 (br, -SiCH<sub>3</sub>-, 3H).

### Film preparation

The terpolymer thin films were prepared by spin-coating from solutions dissolved in MIBK with a concentration of 20 wt %. The silicon wafer pre-treated with piranah solution (H<sub>2</sub>SO<sub>4</sub>/H<sub>2</sub>O<sub>2</sub> 70/30 by v/v) was typically spun at 2000 rpm for 30 s to yield film thicknesses around 500 nm. The coated



**Figure 1** Schematic illustration of the sol-gel polymerization of PMSSQ terpolymer with 1-(4-trimethoxysilylpropoxyphenyl)adamantane (TMSPPA), methyltrimethoxysilane (MTMS), and 1,2-bis(trimethoxysilyl)ethane (BTMSE).

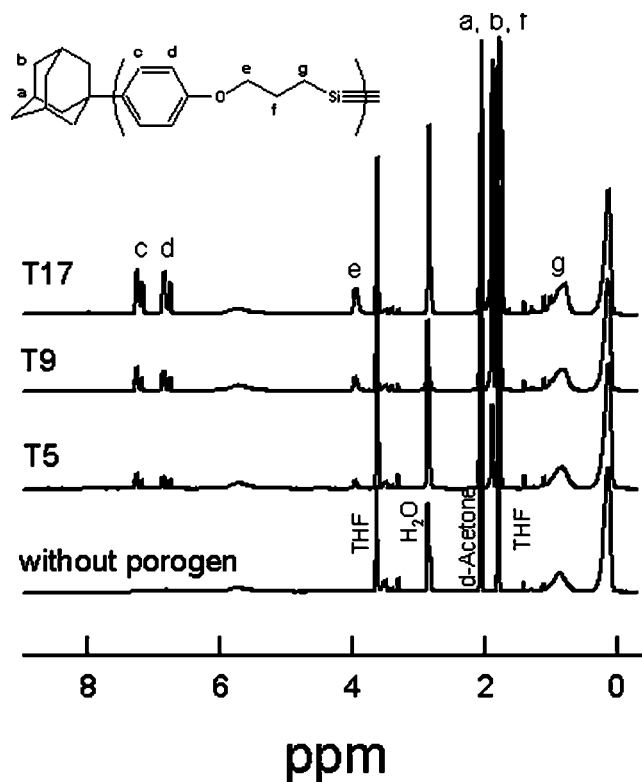
films were then cured at a desired set temperature for 2 h after preannealing the film at 250°C for 30 min with a home-made furnace under N<sub>2</sub> purge.

### Measurements

<sup>1</sup>H NMR spectra were obtained at room temperature using a Bruker (AVANCE) 500 MHz. The NMR spectra were taken in deuterated chloroform for monomers and in acetone-d<sub>6</sub> for the terpolymers. Gel permeation chromatographic (GPC) analyses were carried out using Spectra System P1000 and LR125 Laser Refractometer as a transducer. THF was used as an eluent at a flow rate of 1.0 mL/min and the GPC result was calibrated with polystyrene standards (Polymer Standard Service), which range from 500 to 1,000,000 in molecular weight. Thermal gravimetric analysis (TGA) was performed using a TA model 2050 TGA instrument under N<sub>2</sub> flow at a heating rate of 10°C/min. The solid samples were heated from 50 up to 900°C. The weight loss of the samples (TG curves) was collected. FTIR measurements were performed on a JASCO FT/IR 200 spectrometer. Baseline corrected infrared spectra were obtained for films on silicon wafers in absorbance mode at room temperature. Nitrogen sorption analyses were performed on a Micromeritics ASAP 2010 gas sorption porosimeter. The refractive indices of thin films were measured with a variable-angle multi-wavelength ellipsometer (Gaertner, L2W16C830) with two wavelengths at 633 and 834.5 nm and 49 points wafer scanning set-up. Mechanical properties of all the PMSSQ films were measured by a nanoindenter (Nano Indenter<sup>®</sup> XP, MTS) in the continuous stiffness measurement (CSM). The dielectric constants were measured in the metal-insulator-metal (MIM) configuration, with evaporated aluminum electrodes at 1 MHz using a HP4284 LCR meter. Field emission scanning electron microscopy (JEOL 6330F) was used to investigate the cross-sectional structure of the thin films. Spin coated films were cut into small pieces and fixed vertically on a sampling holder to analyze the internal structure. To minimize the film damage by electron beam and to obtain clear images, Au was sputtered onto the thin films.

### RESULTS AND DISCUSSION

For the synthesis of organosilane compound having porogen, 1-bromoadamantane reacted with phenol to give a high yield (96%) of 4-(hydroxyphenyl)adamantane according to the previously reported method.<sup>17</sup> The corresponding adamantylphenol reacted with allyl bromide in the presence of potassium carbonate as an acid acceptor to give 1-(4-allyloxyphenyl)adamantane in 89% yield. The subsequent conversion of allyl groups was obtained with trimethoxysilane through hydrosilylation reaction

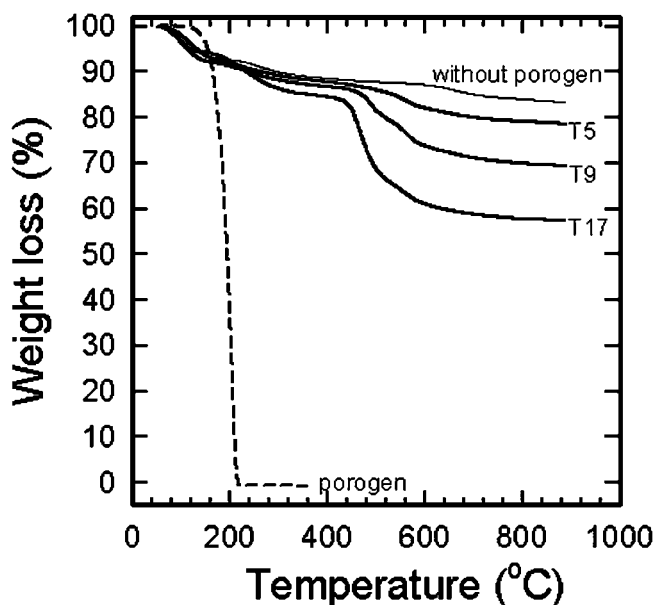


**Figure 2** <sup>1</sup>H NMR spectra of PMSSQ polymers with and without porogen.

under conditions as reported to give 95% yield of 1-(4-trimethoxysilylpropoxyphenyl)adamantane.<sup>18</sup> Subsequently, porogen-containing 1-(4-trimethoxysilylpropoxyphenyl)adamantane (TMSPPA) was polymerized with MTMS and BTMSE to give terpolymers with a variety of porogen content.

The procedure of polymer synthesis was illustrated in Figure 1. For the successful sol-gel polymerization of PMSSQ, the synthetic parameters such as concentration, reaction time, and temperature needs to be controlled. Besides these parameters, it would be more essential to adjust the molar ratio of catalyst to total organoalkoxysilane ( $R_1$ ), the molar ratio of water to total organoalkoxysilane ( $R_2$ ), and the reaction time. In order for the synthesized PMSSQ to be soluble and reproducible,  $R_1$  was controlled to be 0.03 and  $R_2$  was adjusted to range from 3 to 10. Reaction time was also carefully chosen to avoid further gelation during the polymerization.

The chemical structures and the molecular compositions of terpolymer were analyzed with <sup>1</sup>H NMR spectra, as seen in Figure 2. The chemical shifts due to the phenyl group appear at around 7.26 and 6.85 ppm, in addition to the peak assigned to adamantane at about 1.89 ppm. The bridged ethane protons corresponding to BTMSE are also shown at around 0.83 ppm, which is partially overlapped with the peak of the methyl groups of propyl carbons bound

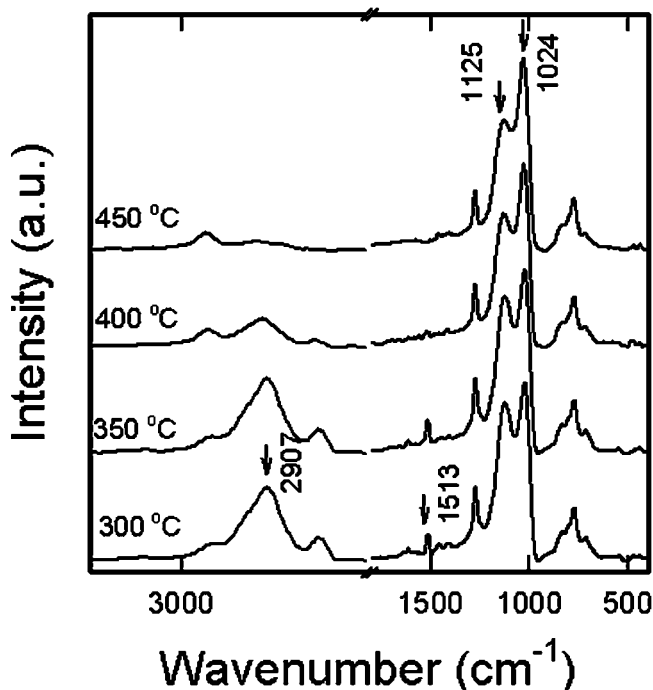


**Figure 3** TGA scans of PMSSQ terpolymers, PMSSQ without porogen, and adamantylphenol template measured under  $N_2$  with a heating rate of  $10^\circ\text{C}/\text{min}$ .

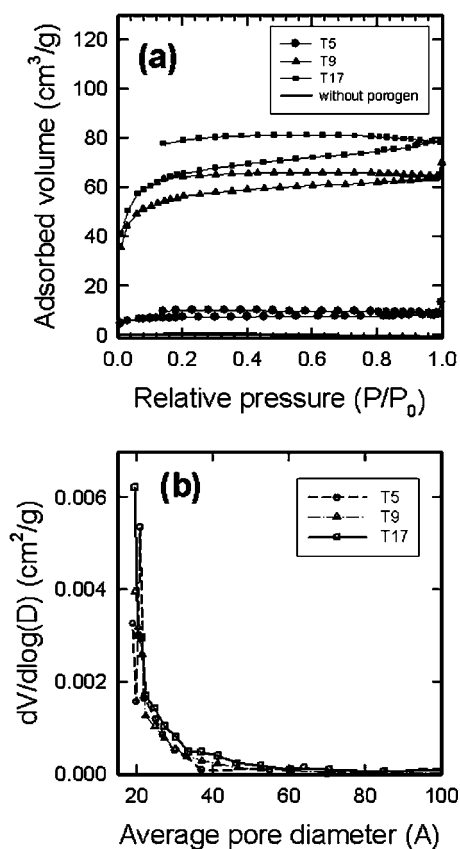
to the silicon atom at about 0.85 ppm. The chemical shifts of Si—OCH<sub>3</sub> and Si—OH are also shown at 3.48 and about 5.74 ppm, respectively, which are similar to the results of PMSSQ without porogen as a reference. Based on the peak area assigned to the phenyl group at 7.26 ppm, the porogen content incorporated into each terpolymer was 5.2%, 8.7%, and 17.4% by mole, which corresponds to 11.4%, 18.3%, and 33.1% by volume, respectively. It is also noted that all terpolymers as well as PMSSQ without porogen include BTMSE of about 20 mol %. Thus, <sup>1</sup>H NMR spectra confirms that adamantylphenol porogen and BTMSE additive was covalently introduced into the PMSSQ matrix. The synthesized terpolymers were denoted T5, T9, and T17, respectively, with increasing porogen. The terpolymer molecular weights and distributions are very important because of their relationship with the thermal and mechanical properties of the low- $\kappa$  film. The weight average molecular weights and distributions of terpolymers were determined using gel permeation chromatography. The molecular weights of the terpolymers decreased with increase of porogen content from T5 (6070 g/mol), T9 (4101 g/mol) to T17 (3690 g/mol) and were lower than that of PMSSQ without porogen (7440 g/mol), which suggests that the steric hindrance effect by the introduction of bulky porogen usually retards the condensation rate, leaving unreacted Si—OH groups during the sol-gel reaction. Thus, the Si—OH contents of T5 (23.66%), T9 (24.93%), and T17 (22.56%) are significantly higher than that (18.65%) of copolymer without porogen.

To investigate the pore forming capability of terpolymers through the thermal decomposition prior to the formation of porous thin film, the thermal decomposition behavior of terpolymers was measured with thermal gravimetric analyzer (TGA) under nitrogen atmosphere. As can be seen in Figure 3, the subsequent weight loss occurred from about  $250^\circ\text{C}$  to around  $600^\circ\text{C}$ . Moreover, as the porogen loading increased, the weight loss in this temperature range also enhanced. In contrast, the PMSSQ without porogens did not show such a massive weight loss above  $400^\circ\text{C}$ . Thus, the mass loss in this step reflects the decomposition of adamantylphenol porogen. The low molecular weight adamantylphenol template also showed the complete weight loss just above  $200^\circ\text{C}$ . On the other hand, the weight loss of porogens in terpolymers shifted to a temperature above  $400^\circ\text{C}$ , indicating that the adamantylphenol groups are chemically linked to the PMSSQ matrix and good pore forming candidates for low- $\kappa$  insulator. For all polymers, initial weight loss up to  $200^\circ\text{C}$  was observed, which are mainly associated with the loss of water generated during polycondensation reaction of silsesquioxane material as well as the loss of residual solvent, as reported elsewhere.<sup>19–21</sup>

Thermally activated loss of porogens was further monitored by FTIR spectroscopy. Figure 4 shows the typical change of infrared spectra of T17 terpolymer thin film with different treatment temperatures. IR band of C—H stretching vibration at  $2907\text{ cm}^{-1}$  as



**Figure 4** The change in the FTIR spectra of T17 terpolymer film with treatment temperature.



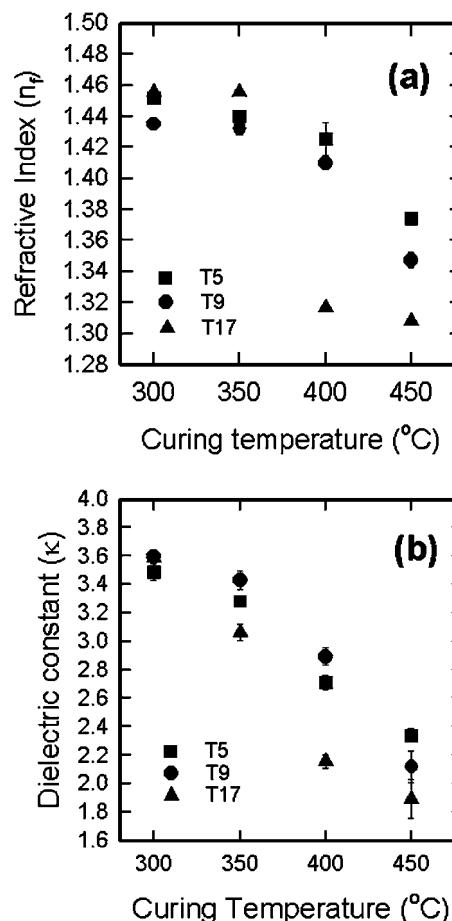
**Figure 5** (a)  $N_2$  adsorption isotherms and (b) pore size distribution curves of terpolymer samples treated at  $450^\circ\text{C}$  under  $N_2$ .

well as a peak at  $1513\text{ cm}^{-1}$  due to the  $\text{C}=\text{C}$  of the phenyl group reflects the expected adamantylphenol group. Similarly, an IR band at  $1407\text{ cm}^{-1}$  corresponds to the  $\text{C}-\text{H}$  deformation vibration of the ethane bridges of BTMSE.<sup>22,23</sup> The strong broad band which appeared from  $1000$  to  $1200\text{ cm}^{-1}$  was assigned to the  $\text{Si}-\text{O}-\text{Si}$  of silsesquioxane backbone in addition to the peak due to  $\text{Si}-\text{C}$  at  $774\text{ cm}^{-1}$ , respectively. Infrared spectrum with thermal treatment reveals that the peak intensity at  $2907\text{ cm}^{-1}$  notably decreases along with vibration at  $1513\text{ cm}^{-1}$ , while the ethane bridge of BTMSE at  $1407\text{ cm}^{-1}$  and  $1271\text{ cm}^{-1}$  still remains undamaged. When treated at  $450^\circ\text{C}$ , the porogen nearly disappears, leaving residual carbon-like materials at about  $2900\text{ cm}^{-1}$ . In contrast, a small amount of the BTMSE bridged structure at  $713$  and  $1407\text{ cm}^{-1}$  is still retained with the reduced intensity relative to that of the previous treatment. Upon treatment, the ratio of the peak intensity corresponding to the cage-like structure at  $1125\text{ cm}^{-1}$  to that due to the networked at  $1024\text{ cm}^{-1}$  decreased from 0.81 to 0.66, which indicates that the film structure becomes denser owing to the structural rearrangement.

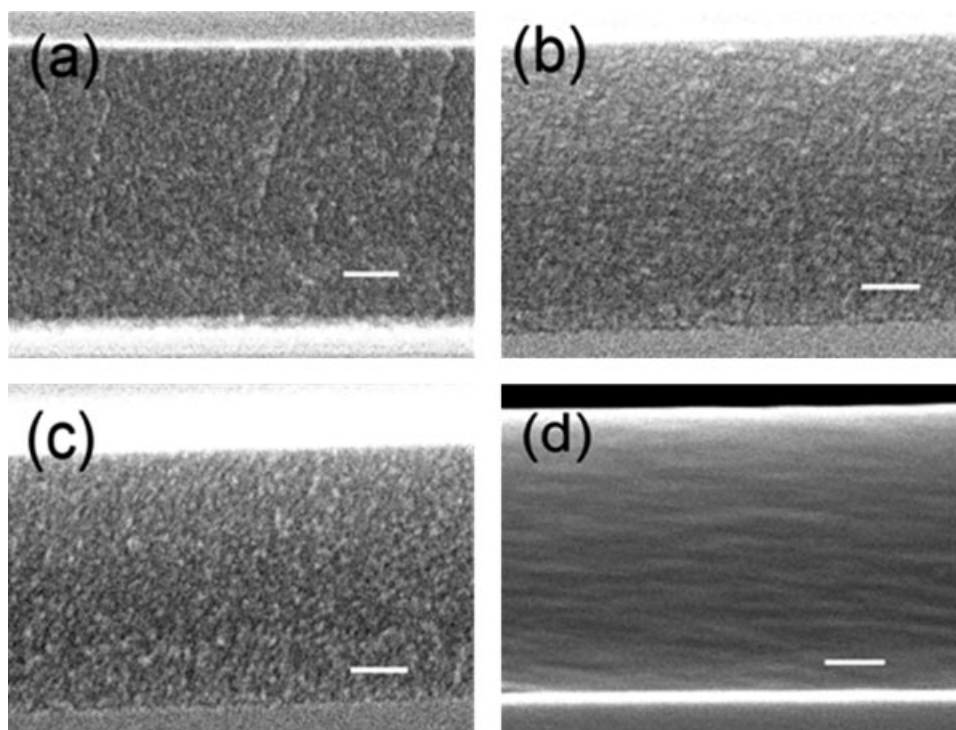
Figure 5 shows nitrogen adsorption/desorption isotherms and pore size distribution curves of terpol-

ymers. For the sorption isotherm measurements, spin-coated films were initially treated at  $450^\circ\text{C}$  and were scraped off the substrates to yield powder samples. It was found that there is no adsorption for PMSSQ without porogen, showing the  $0.5\text{ m}^2/\text{g}$  of very low BET surface area. In contrast, BET surface area of T5, T9, and T17 terpolymers increased from  $24.6\text{ m}^2/\text{g}$  and  $194.2\text{ m}^2/\text{g}$  to  $227.6\text{ m}^2/\text{g}$  by increasing porosity, indicating that the nanoporous structure was formed by the degradation of porogens. In addition, isotherms of all terpolymers have non-closed hysteresis. These characteristics are fairly related to pores of ink-bottle type due to very low desorption of nitrogen in a low relative pressure region.<sup>24–26</sup> BJH pore size distribution curves also indicates that all films have microporous characteristics without killer pores above  $50\text{ \AA}$ , confirming that the nanoporous structure of all films dominantly consisted of pores below  $50\text{ \AA}$ .

Figure 6 shows the change of refractive index ( $n_f$ ) of thin films at different treatment temperatures. The refractive index values for all films decreased with



**Figure 6** The change of (a) refractive indices and (b) dielectric constants with treatment temperature and porogen content.



**Figure 7** Cross-sectional FE-SEM micrographs of (a) T5, (b) T9, (c) T17, and (d) copolymer prepared without porogen. Scale bar denotes 100 nm.

the treatment temperatures. For instance, the refractive index of T17 was lowered from  $1.46 \pm 0.01$  to  $1.31 \pm 0.01$  upon treatment temperature. The reduction of refractive index is also proportional to the porogen content incorporated, indicating that the decomposition of adamantylphenol porogen effectively facilitates the formation of nanopores within the films. The additional contribution in reduction of refractive index can be expected from the removal of silanol groups through polycondensation along with the removal of phenyl groups. The relative porosity of a porous film was estimated from the change of refractive indices in both the porous film and fully cured matrix using the Lorentz–Lorenz equation.<sup>27</sup> The calculated porosity values were 8.4%, 17.9%, and 29.5% for T5, T9, and T17 films, respectively, which is lower than the volume fraction of porogen incorporated and believed to be due to the formation of residual carbaceous materials, as can be observed in infrared spectra.

The change of low- $\kappa$  value measured using the MIM (metal-insulator-metal) structure was also similar to that of the refractive index, as illustrated in Figure 6. As a reference, the dielectric constant ( $k$ ) of the PMSSQ film without porogen was 2.9, which is in good agreement with the previously reported value.<sup>28</sup> The dielectric constants of all the films tested decreased with treatment temperature and porogen loading. For instance, the  $k$ -values of T9 and T17 films reduced to 2.12 and  $1.89 \pm 0.14$ , respectively, after

curing at 450 °C. Thus, this reduction in  $k$ -values as well as refractive indices confirms that the nanoporous structure was formed by the decomposition of adamantylphenol porogens. Additionally, it is noted that the addition of BTMSE inhibits the collapse of porous structure observed in our previous study.<sup>14</sup> This means that the mechanical stability of PMSSQ thin film can safely be optimized through the introduction of organosilicate with abundant hydrolysis-condensable groups such as BTMSE without further increase of dielectric constant.

Related to the porous structures of the low dielectric film, the mechanical properties of the films were taken into account because the film should be resistant against harsh conditions such as the chemical mechanical planarization (CMP) process. The elastic modulus was measured with a nanoindenter in dynamic contact mode (DCM). All the reported values were read at 50 nm of the displacement depth, which are about 1/10 of the total film, with thickness enough to neglect the substrate effect. After treatment at 450 °C, the elastic modulus of films reduced from  $9.2 \pm 0.03$  (T5),  $3.8 \pm 0.06$  (T9) to  $3.7 \pm 0.04$  GPa (T17), respectively. The surface hardness behaviors of the films were also similar to that of the modulus, where surface hardness of T5 and T17 films were  $1.2 \pm 0.02$  and  $0.4 \pm 0.01$  GPa, respectively. These results are dominantly due to the formation of the porous structure by the decomposition of porogen.

The cross-sectional FE-SEM photographs for the thin films are shown in Figure 7. The PMSSQ film prepared without porogen clearly shows a very smooth surface without noticeable texture variation, as reported previously.<sup>15</sup> In contrast, all the terpolymer films have a clear texture variation due to the difference in electron density between the pores and the matrix. Pores in thin films also seem to be isolated, showing a pore size of around 10 nm within the resolution of FE-SEM. Thus, it is confirmed that the decomposition of adamantane porogen leads to the formation of nanoporous structure. Indeed, it was expected that pore size or pore interconnectivity of films would be increased with porogen content. However, the observed SEM data indicate that there was no clear variation of the pore morphology with porogen increase. Nevertheless, T17 film porosity was observed to be higher than that of T5 and T9, as supported by the BET result, although detailed understanding about porous structure remains to be a challenge.

### CONCLUSIONS

In the present study, PMSSQ terpolymers composed of chemically linked adamantylphenol porogen, BTMSE, and MTMS were successfully synthesized and their thin film properties were investigated, for low-k applications, as well as the change of molecular structures with thermal treatment. Mechanically stable nanoporous low-k thin film was successfully achieved with a k-value of 1.89 and a modulus of 3.7 GPa by the decomposition of adamantylphenol porogen and the introduction of BTMSE. Finally, this strategy is believed to be very useful for designing nanoporous organic and inorganic thin films with potential applications ranging from low-k dielectrics to antireflection coating materials to pore-based sensing devices.

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